U.S. DEPARTMENT OF COMMERCE
National Technical Information Service

PB-256 368

Laboratory Procedure for Determining the Total Heat of Combustion in Solid Wastes

Environmental Protection Agency

1971

236034

PB 256 368

LABORATORY PROCEDURE FOR DETERMINING THE TOTAL HEAT OF COMBUSTION IN SOLID WASTES

A Division of Research and Development Open-File Report (RS-03-68-17)

U.S. ENVIRONMENTAL PROTECTION AGENCY

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NATIONAL TECHNICAL
INFORMATION SERVICE
U. S. DEPARTMENT OF COMMERCE
SPRINGFIELD, VA. 22161

BIBLIOGRAPHIC DATA SHEET	1. Report No.	2.	3. Recipient's Accession No. PB-256 368
4. Title and Subtitle Laboratory Procedure for Determining the Total Heat of Combustion in Solid Wastes			5. Report Date 1971 6.
7. Author(s) Donald L. Wilson			8. Performing Organization Rept. No.
9. Performing Organization Name and Address Environmental Protection Agency Cincinnati, Ohio Solid Waste Management Office			10. Project/Task/Work Unit No. EPA-RS-03-68-17 11. Contract/Grant No.
12. Sponsoring Organization Name and Address			13. Type of Report & Period Covered 14.

15. Supplementary Notes

16. Abstracts

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17. Key Words and Document Analysis. 17a. Descriptors

*Solid waste disposal,
*Heat of Combustion,
*Measurement,

Calorimeters,
Instrumentation,
Experimental design,
Laboratory equipment,
Standards,
Incinerators.

17b. Identifiers/Open-Ended Terms

17c. COSATI Field/Group 7 D 13 B

18. Availability Statement National Technical Information Service Springfield, Virginia 22161	19. Security Class (This Report) UNCLASSIFIED	21. No. of Pages 26p
	20. Security Class (This Page UNCLASSIFIED	22. Price 4.00/2.25

NTIS-35 (REV. 10-73) ENDORSED BY ANSI AND UNESCO. THIS FORM MAY BE REPRODUCED USCOMM-DC 8265-P74

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22. **NTIS Price.** Leave blank.

LABORATORY PROCEDURE FOR DETERMINING THE TOTAL HEAT OF COMBUSTION IN SOLID WASTES

A Division of Research and Development
Open-File Report (RS-03-68-17)

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U.S. ENVIRONMENTAL PROTECTION AGENCY
Solid Waste Management Office
1971

In publishing an analytical method which employs specific brands of laboratory supplies and instrumentation, the U.S. Environmental Protection Agency does not imply that other commerical products could not be utilized with appropriate modifications in the procedure. This method is meant to be used in conjunction with the Technical Manual No. 130, Parr Instrument Company, and was written as such.

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DISCUSSION

The heats evolved in the complete combustion of many compounds in oxygen have been carefully determined. The method ordinarily used is to burn the substance in a combustion bomb and to measure the heat evolved.

The term "heat of combustion" refers to the amount of heat liberated per unit of substance burned. This process involves the change in enthalpy or heat content, H, of the system. The heat of combustion or ΔH_c described in this method is expressed in calories per gram of sample or British Thermal Units (Btu) per pound (#) of sample.

The heat contents of various solid waste materials are important to some of the volume reduction processes used to dispose of waste. In the incineration process, for example, the operating efficiency of an incinerator can be measured by energy balance techniques, and analyzing heat contents of the solid wastes before and after incineration is essential. Also the heat value is an important characteristic of solid waste needed for incinerator design. Performing these same heat content analyses on incinerator residue and compost used for landfill is also essential because the stability of these waste products is a function of their heat contents.

To determine directly the heat change involved in a reaction calorimeters are employed. A calorimeter consists essentially of an insulated container filled with water in which is immersed the reaction chamber. In an exothermic reaction the heat generated is transferred to the water, and the consequent temperature rise of the water is read from an accurate

thermometer immersed in it. Knowing the quantity of water present, its specific heat, and the change in temperature, the amount of heat evolved in the reaction may be calculated. Special corrections must be applied for radiation, rate of cooling of the calorimeter, temperature rise of the vessels, stirrer, etc. To compensate for these corrections the heat capacity of the calorimeter is determined by burning a definite amount of a standard.

This method describes the capabilities and limitations of a Parr Adiabatic Calorimeter, and furnishes the instructions needed to obtain best results with this apparatus. Using this method the heats of formation of nitric acid and sulfuric acid are involved in the total heats of reaction; therefore, they are determined and the data is adjusted.

APPARATUS

Requirements

1. Balance, analytical, 150 g capacity, 0.1 mg readability
2. Beakers: one-30 ml, one - 100 ml, one - 250 ml
3. Bottle, Aspirator, plastic, 1 gallon capacity
4. Bottle, Carboy, 5 gallon capacity
5. Bottle, Reagent, 1 liter capacity
6. Bottle Wash, plastic, 125 ml capacity
7. Bulb, Rubber, 30 ml capacity
8. Buret, Clamps
9. Buret, 50 ml capacity, three-way stopcock

10. Calorimeter, Adiabatic, Parr No. 1221, with Technical Manual No. 130
11. Calorimeter Bomb, double valve, Parr No. 1101 (Fisher #4-392-1)
12. Calorimeter Fuel Capsule, stainless steel, Parr No. 43AS, (Fisher #4-385-5); eight or more
13. Calorimeter Ignition Unit, transformer type, Parr Series 2900
14. Calorimeter Mercurial Thermometers, Parr No. 1601; two
15. Calorimeter Oxygen Filling Connection, safety type, Parr Series1823
16. Calorimeter Thermometer Reading Lens, Parr No. 3003; two
17. Calorimeter Water Heater, automatic, electric Parr Series 1500
18. Carver Laboratory Press, Model C, or equivalent, (Will #22562)
19. Carver Test Cylinder Outfit or Dies, "specially made," internal diameter of cylinder 3/4 inch, length of internal cylinder chamber 3 1/4 inches, similar to outfit with 1 1/8 inch internal diameter of cylinder (Will # 22591)

    NOTE: Although the smaller die is preferred, a larger die may be used with modifications in the procedure. The pellets are made thicker and broken into four or more parts by using a sharp object or breaking by hand. Handle the pellets as little as possible.
20. Clamp, pinchcock
21. Desiccator
22. Dish, Weighing,: with lid; eight or more
23. Drying Oven
24. Flasks, Volumetric: one - 2 liter, one - 1 liter
25. Funnels-Filling: one - 80 mm diameter, one - 250 mm diameter

26. Jack, laboratory
27. Pipet, volumetric, 1 ml capacity
28. Ring, Support, with clamp, four-inch outside diameter
29. Sissors
30. Stopwatch
31. Support Stand, rectangular base, 24-inch rod; two
32. Tubing, glass, 1/2-inch I. D., about three feet
33. Tubing, plastic, 1/4-inch I. D. ; about three feet
34. Tubing, plastic, 1/2-inch I. D.; about six feet

Assembling

The analyst is referred to the Parr Company's Manual No. 130, pages 17-21, for the general arrangement of the calorimeter and accessories. The analyst is instructed in the procedure section when to plug in the water heater cord.

For convience, the distilled water is stored in a 5 gallon-carboy-bottle with a glass tubing through the lid to near bottom. A plastic tube is connected to this glass tubing and a pinchcock clamp is used to prevent the flow of water. The sodium carbonate solution is stored in a 1 gallon plastic bottle with aspirator. Plastic tubing, 1/4-inch I. D., connects the aspirator to the automatic filling buret. The plastic bottle rests upon a laboratory jack atop a shelf, thus allowing the level of reagent to be higher than the solution within the buret. A 250 ml beaker is put beneath the buret to prevent spilling. A support ring is connected

to a support stand, about one foot above the base. The one ml pipet stored in an inverted filling funnel, 80 mm in diameter. For ease of pipeting, some distilled water is stored in 30 ml beaker.

REAGENTS

Chemical Requirements

1. Benzoic acid, primary standard with calorific value known, (Fisher #A-68)
2. Methyl orange, ACS grade, (Fisher #M-216)
3. Oxygen, 99.5 percent pure (the oxygen should be prepared from liquid air. Oxygen, prepared by electrolysis, contains traces of hydrogen.)
4. Sodium carbonate, Na_2CO_3, ACS grade, anhydrous
5. Steel wool
6. Wire, nickel-iron-chromium alloy, No. 34 B. & S. gauge, wound on a card 10 cm long, (Parr 45 C10)

Preparation

1. Sodium carbonate solution, 0.0725N: Dissolve 3.8421 g of Na_2CO_3 in distilled water and dilute to one liter. (Store in plastic bottle with aspirator)
2. Saturated methyl orange solution: Dissolve 0.5 g of Methyl orange in distilled water and dilute to one liter. (Store in reagent bottle)
3. Wash solution: Pipet 1 ml of saturated Methyl orange solution into a one-liter flask and dilute to the liter mark with distilled water.

4. Ignition wire: Using 10 cm marks on card provided with wire, measure and, with sissors, cut off 10 cm of wire. Several 10 cm lenghts may be made at one time and stored in 100 ml beaker.

SAFETY PRECAUTIONS

The analyst is referred to the Parr Company's Manual No. 130, page 9, "Hazards of Operation".

SAMPLE PREPARATION

The details of total sample preparation procedures which describe the drying and grinding techniques are not discussed in this method. In general, since this method restricts the quantity of sample analyzed to be about 1.0 g or less and thoroughly dried, the entire sample (except glass, metals, and ceramics) must be ground until the particle size is reduced to less than 2 mm, dried until no more loss in weight, and then completely mixed before being analyzed.

Fluffy materials (standard Benzoic Acid, raw refuse, and compost) must be pelletized in order to fit the metal capsules. The procedure for pelletizing is as follows:

Procedure	Comments
1. Using Carver Laboratory Press with 3/4-inch diameter punch and die, loosly fill the chamber 1/2 to 3/4 full of sample.	1. A 1 1/8-inch diameter punch and die may be used; however, the pellet will have to be broken. If at all possible the 3/4-inch punch and die should be used.

2. Compress the sample until the pressure is 5000 to 7000 lbs., as read by the dial.

3. Transfer the pellet to a weighing dish with lid.

4. Repeat for each determination.

3. Touch pellets as little as possible with hands.

4. See "Method Evaluation".

PROCEDURE

The following procedure applies to all solid waste materials and the Benzoic Acid standard.

In order to ensure good precision and not to exceed the capacity of the instrument, the analyst is advised to follow these restrictions:

Sample type	Sample weight	Specifics of Analysis
Benzoic Acid	1.0 ± 0.2 g	pelletized
Raw Refuse	0.4 to 0.8 g	pelletized
Residue (Combustible)*	0.4 to 0.8 g	pelletized
Residue (Fines)**	0.8 to 1.2 g	combustion aid added
Fly Ash	0.8 to 1.2 g	combustion aid added
Compost	0.4 to 0.8 g	pelletized

*Mostly readily combustible materials which remained on a 1/2-inch sieve during manual sorting at the incinerator site.

**Material remaining after combustibles, metal, glass, and ceramics removed from incinerator residue.

Procedure	Comments
1. Weigh sample into tared capsule. If pelletized pellet must be thin enough to keep the weight within restrictions. If a combustion aid is added, sandwich the sample evenly between pellets of the combustion aid, keeping the total weight of the combustion aid in excess of the sample weight.	1. a) Capsules should have previously been cleaned with steel wool and water. Then dried and stored in desicator until weighed. b) Use analytical balance and record to the fourth decimal place. c) Duplicate or triplicate samples are needed (See Evaluation of Method). d) Combustion aid is pelletized Benzoic Acid. e) Remember: Not more than 10,000 calories should be liberated in any one test. f) If the combustion aid is made with a die larger than the recommended size, the pellet must be broken to meet restrictions. Keep the larger pellet under all of the sample.
2. Bind the fuse wire to 4A and 5A electrodes.	2. See Parr Manual No. 130, page 28.
3. Place the capsule with sample in 5A electrode.	3. The fuse wire should be bent so that a loop is just <u>above</u> the sample.
4. Pipet one ml of distilled water into the No. 1101 bomb.	4. Use a rubber bulb for the pipeting. (Distilled water stored in 30 ml beaker.)

5. Place bomb head into the cylinder.	5. a) Make certain sealing ring is in good condition. b) Always keep cylinder in upright position and do not disturb the sample.
6. Screw cap down firmly by hand.	6. a) Keep outlet needle valve open. b) Parr Manual, Fig. 4, page 11, may be helpful.
7. Close outlet needle valve.	
8. Remove inlet valve thumb nut.	
9. Attach filling tube with union nut firmly, by hand.	9. Use Parr, Series 1823. Consult Parr Manual, pages 20 and 21.
10. Open the filling connection control valve slowly.	10. a) Oxygen tank valve must be open. b) If opened quickly, sample may come out of capsule, thus causing incomplete combustion.
11. Allow pressure to rise slowly until gauge reads 30 atmospheres.	11. Although pressure range, according to manual, is 25 to 35 atm., same pressure must be used for all test.
12. Close connection control valve.	
13. Push sideways on ball knob under relief valve.	13. Relieves gas pressure in connecting tube.
14. Detach connecting tube.	
15. Replace thumb nut.	

16. Place bomb with its feet spanning the boss in the bottom of the bucket.	
17. Attach the thrust terminal to the bomb.	17. Lead wire should not extend above the bucket.
18. Lower bucket into the jacket with stirrer at rear.	18. Lower handle to back of bucket.
19. Place 2000 ml (about 2000g) of distilled water, at room temperature, into the bucket.	19. a) Keep the amount of water constant throughout all tests, once standardization is completed. b) A two-liter volumetric flask should be used.
20. Close the calorimeter by swinging the cover to the right and lowering it with the cam lever.	20. Parr Manual, figures 8 and 9, page 13, may be helpful.
21. Make <u>SURE</u> the pump and stirrer drive shafts are seated properly. This is done by seeing that the pump and stirrer pulleys are down as far as possible and move freely.	21. If the water in the bucket is not being stirred, the temperature of this water will decrease soon after the final increase due to the sample combustion.

22. Lower the thermometers into the calorimeter.

22. The thermometers should be submerged about the same distance as during standardization.

23. Having all valves before and after the water heater open, turn on the water slightly. Note: The two valves nearest the calorimeter are always kept completely open. NEVER close all four valves completely.

23. a) If the water is turned on too much leaks may occur.
b) See Parr Manual No. 130, pages 18 and 19.
c) Counter-clockwise turns open all valves.

24. Wait until water runs from the discharge tube, then close the hot water valve and plug in the water heater.

24. CAUTION: The water flow and heating of the water continues throughout the use of the instrument; however, the water heater may become overheated if its electric cord is not occasionally disconnected for a few minutes.

25. Start the stir motor and again wait until the water runs from the discharge tube. (Occasionally the pulleys need to be tapped down to maintain circulation of the water.)

26. Adjust the cold and hot control valves so that the jacket temperature, "left" thermometer, is the same or slightly lower than the bucket temperature, "right" thermometer.

26. a) The main water valve may need adjusting. Lowering the flow rate increases the control over water temperature.

b) Read thermometers to nearest 0.005 F.

c) See Parr Manual No. 130, pages 14 and 15.

d) Occasionally, check the thermometers for mercury separations and reunite the mercury, if necessary, in accordance with the instructions given on the thermometers certificates.

27. If no change in the bucket temperature after one or two minutes, record this temperature.

27. Use reading lenses and record to nearest 0.005 F.

28. Press button on ignition unit and stopwatch at the same time.

28. Keep switch closed for 4 to 5 seconds <u>only</u>.

29. During the rapid rise in bucket temperature, usually 5 minutes, keep the jacket temperature about the same or slightly lower than the bucket temperature.

29. The rapid rise in bucket temperature begins about 20 seconds after button is pressed.

30. When the system is approaching final equilibrium temperature, keep the jacket temperature within 0.1 degree of the bucket temperature.

31. Record the final maximum bucket temperature.

31. a) The stable maximum temperature is when the same temperature is observed in <u>three</u> successive readings.

b) Use reading lenses and record to nearest 0.005 F.

32. With the cold water valve slightly open, close the hot hot water valve.

32. This will allow the temperature of the jacket water to lower to a starting temperature for the next analysis.

33. Stop the stirr motor and wait for water to drain out of the cover.

33. Water has drained from the cover when the flow from the discharge tube returns to normal.

34. Raise the thermometer support with thermometers, to its top position.

34. This should remain "UP" at all times while the cover is off.

35. Using cam lever, lift cover and swing to left.

36. Lift the bucket out of the jacket, disconnect the thrust terminal wire, and remove the bomb.

37. With a filling funnel, pour the water from the bucket into the 2-liter flask.

37. A 250 mm diameter funnel is used.

38. With sponge, paper towel or napkin, remove excess water from top of bomb.

39. Slowly relieve the pressure from the bomb by opening outlet needle valve.

39. This step should require about 1 to 3 minutes.

40. Remove screw cap and bomb head.

40. Discard test if <u>definite</u> evidence of incomplete combustion.

41. Using wash bottle with prepared solution, wash bomb head and all interior surfaces of bomb, then collect the washings in a 250 ml beaker. Carefully lay the bomb head aside.

41. The bomb head and all interior surfaces should be rinsed with distilled water and allowed to drain before the next analysis.

42. Titrate the washings to a reagent color end point, using 0.0725 N sodium carbonate solution and record ml of titrant.

42. a) Record ml titrant as calories, to nearest tenth, involved in heat of formation of nitric acid.

b) Most of the time this value represents less than 5% of the final answer. However, if it represents more, save the solution for determining sulfur content, as directed by the Parr Manual No. 130, pages 37, 48 and 49.

43. Carefully remove all unburned pieces of fuse wire from the bomb head and measure their combined lengths with the card originally containing the wire.

44. Record the calorific value, to nearest tenth, of wire used.

45. Repeat steps 1 through 24 for each analysis.

45. Steps 23 and 24 are not repeated in continuous operation.

46. At the end of each continuous operating cycle, disconnect the electric plug of the water heater and then turn off the main source.

STANDARDIZATION

The term "standardization" denotes the determination of the energy equivalent or water equivalent factor (W) of the system. (See Parr Manual No. 130, page 23.) Benzoic Acid, primary standard with calorific value known, is the material used to determine the W factor. The benzoic acid is analyzed in the manner previously described under "Procedure."

CALCULATIONS

Standards

Compute the energy equivalent by substituting in the following equation:

$$W = \frac{\Delta H_c M + e_1 + e_3}{t}$$

where:

W = energy equivalent of calorimeter in calories per degrees Fahrenheit

ΔH_c = heat of combustion of standard benzoic acid in calories per gram

M = mass of standard benzoic acid sample in grams

t = corrected * temperature rise in degrees F

e_1 = correction for heat of formation of nitric acid, in calories

e_3 = correction for heat of combustion of firing wire, in calories

*Temperatures are corrected for thermometer variations which are from graphs furnished by the Parr Company for each thermometer. However, if sample has heat content above 100 or 200 calories per gram, this correction will have little effect upon the final answer.

Samples

Compute the calorific value per gram of sample by substituting in the following equation:

$$\Delta H_c = \frac{tW - e_1 - e_2 - e_3 - e_4}{M}$$

where:

ΔH_c = heat of combustion of sample in calories per gram*

t = corrected temperature rise in degree F

W = average energy equivalent of calorimeter found by standardization, in calories per degree F

e_1 = correction for heat of formation of nitric acid, in calories

e_2 = correction** for heat of formation of sulfuric acid, in calories

e_3 = correction for heat of combustion of firing wire, in calories

e_4 = heat of combustion of combustion aid, in calories

M = Mass of sample in grams, should not include Mass of combustion aid aid

*(ΔH_c cal/g)=(1.8) ΔH_c Btu/lb

**Correction is not necessary if considered in e_1 and e_1 is not more than 5% of the final answer.

METHOD EVALUATION

The accuracy of this method was established by analyzing sucrose, NBS #17. The average of three determinations was 3981 cal/g which is 46 cal/g or 1.2% from the true value.

This method can analyze solid wastes materials to within 40 Btu/#, depending upon the type* of sample; therefore, this method should not be employed to analyze samples which contain less heat content than the pooled standard deviation for that type of sample (Table 1). To ensure good precision the particle size of the samples must be less than 2mm or pass through a #60 sieve, then the sample must be thoroughly mixed before being analyzed.

The standard deviation of the "W: factor, on 5 observations, was 10 cal per degree F for one analyst in 1968 and 6 cal per degree F for another analyst in 1970.

*Types of solid wastes in this method refer to only solid samples (domestic origin) as: raw refuse, incinerator fly ash, incinerator residue, and compost.

TABLE 1

STANDARD DEVIATION[a] OF THE Btu/# DETERMINATION ON SOLID WASTES

Type of Sample	No. of samples	Btu/# Duplicates	Btu/# Triplicates	Btu/# Range
Raw Refuse	10	57[d]	40	7366 to 9999
Residue (Fines)[b]	7	76	53	249 to 2470
Residue (Combustibles)[c]	7	76	54	4457 to 8182
Fly Ash	7	56	40	Zero to 601
Compost (without sewage sludge)	2	–	173	5661 to 6118
Compost (with 6% sewage sludge)	8	–	227	4504 to 7510
Compost(with 10-15% sewage sludge)	2	–	110	3915 to 4174

[a]A variance estimate can be calculated from the duplicate (or triplicate) set of observations for each sample. The pooled variance is essentially an average of all such estimates for samples of a given type. It is assumed that a single underlying variance exists for all samples of a given type. The pooled variance is then the best estimate of this underlying variance. The pooled standard deviation is the square root of the pooled variance and is used to estimate the underlying standard deviation.

[b]"Fines" are materials remaining after most of the readily combustible substances have been removed by manual sorting. The sorting was performed at the incinerator cites and a 1/2-inch sieve was employed to assist in the separation.

[a]"Combustibles" or "Organics" are mostly the readily combustible materials. Unlike the "Fines" these materials are usually retained on a 1/2-inch sieve.

[b]The absolute value of the difference between duplicated observations should not exceed 1.96 (2) (s), confidence interval, or 158 Btu/#, more than 5% of the time. The covariance between the duplicated observations was ignored.

ACKNOWLEDGMENT

The author gratefully acknowledges the contributions of Mr. Richard Carnes, Mrs. Annella Johnson, Miss Nancy Ulmer, Mrs. Donna Barnett, Mr. Israel Cohen, and Mr. James Doerger for assistance in developing this method.

The author thanks the Division of Technical Operations, Solid Waste Management Office, for the provision of samples from incinerators, and PHS-TVA Compost Plant, Johnson City, Tennessee, for the compost samples.

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